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A GENERAL EQUATION FOR PREDICTING X-RAY INTENSITIES FROM STRATIFIED SAMPLES IN THE ELECTRON MICROPROBE

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PHYSICAL METALLURGY RESEARCH LABORATORIES

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MICROMEDIA

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by

R.H. Packwood and K.S. Milliken

INTRODUCTION

The mean depth of penetration of 10 kV electrons into low atomic nuclear material is of the order of 5×10^{-5} cm. Even so, this is greater than the thickness of some components in LSI (large scale integrated circuit) devices in common use in the electronics industry. These devices cannot be analyzed in the conventional way. However, an extension of the gaussian $\phi(\rho z)$ model may avoid the difficulties that the conventional microprobe theory has with compositional stratification of the analysis volume.

THEORY

The depth distribution of electron excited X-ray production, $\phi(\rho z)$, has been measured experimentally on a number of elements, at different accelerating potentials and X-ray energies.

In 1979, one of the authors (RHP) found that when $\log_e \phi(\rho z)$ was plotted against $(\rho z)^2$, the square of the mass thickness, straight lines were obtained along the ρz-axis with the exception of the near-surface region where transient behaviour is observed.

These findings imply that $\phi(\rho z)$ can be represented by a function of the general type:

$$\phi(\rho z) = \gamma_0 \exp(-\alpha^2 \rho z^2) - (\gamma_0 - \phi_0) \exp(-\alpha^2 \rho z^2 - \beta \rho z) \qquad (1)$$

i.e., a gaussian, modified at small ρz by a 'capacitor charging' function.

To calculate the observed intensity, $\phi(\rho z)$ must be multiplied by an X-ray absorption factor and integrated over all values of ρz, thus;

$$I_{obs} = C_A \int_0^\infty \phi(\rho z) \exp(-\chi \rho z) \, d\rho z \qquad (2)$$

where $\chi = \mu \operatorname{cosec} \theta$ as usual, and C_A the concentration of the element of interest.

For a semi-infinite sample we have as a result

$$I_{obs} = C_A \left[\gamma_0 \exp(\chi/2\alpha)^2 \operatorname{erfc}(\chi/2\alpha) - (\gamma_0 - \phi_0) \exp\left(\frac{\beta+\chi}{2\alpha}\right)^2 \operatorname{erfc}\left(\frac{\beta+\chi}{2\alpha}\right) \right] \qquad (3)$$

where $\operatorname{erfc}(x)$ is the complementary error function given by:

$$\operatorname{erfc}(x) = 1 - \operatorname{erf}(x) \qquad (4)$$

For the general case of a layer inside a sample extending from mass depths, δ' to δ, the observed intensity can be shown (see Appendix) to be given by:

$$I_{obs} = c_A \exp(-\delta' x') \left[\gamma_0 \exp\left(\frac{x}{2\alpha}\right)^2 \left(\operatorname{erf}\left(\alpha\delta + \frac{x}{2\alpha}\right) - \operatorname{erf}\left(\alpha\delta' + \frac{x}{2\alpha}\right) \right) \right.$$

$$\left. - (\gamma_0 - \phi_0) \exp\left(\frac{\beta+x}{2\alpha}\right)^2 \left(\operatorname{erf}\left(\alpha\delta + \frac{\beta+x}{2\alpha}\right) - \operatorname{erf}\left(\alpha\delta' + \frac{\beta+x}{2\alpha}\right) \right) \right] \qquad (5)$$

where x' is the X-ray absorption in the material covering the buried layer.

By putting $\delta' = 0$ and $\delta = \infty$ we have the formula for a semi-infinite sample.

Setting $\delta' = 0$, gives the X-ray intensity from a surface deposit of thickness δ; and δ', $\delta = \infty$, gives the intensity from a substrate covered by of material with absorption factor x'.

The functional variation in the parameters α, β, γ_0 and ϕ_0 are approximately as follows:

$$\gamma_0 \doteq 10\pi \frac{U_0}{(U_0 - 1)} \left(1 + \frac{10}{\log_e U_0} \left(U_0^{-0.4} - 1 \right) \right) \qquad (6)$$

$$\phi_0 \doteq 1 + 2.7\eta \left(1 - \exp\left(\frac{1 - U_0}{2}\right) \right) \qquad (7)$$

$$\alpha \doteq \frac{4.5 \times 10^5}{E_0^{0.75}} \left(\frac{Z - 1.3}{Z} \right) \left(\frac{Z}{A} \right)^{0.5} \left(\frac{\frac{Z}{A} \log_e \left(\frac{1.166}{J} \left(\frac{E_0 + E_c}{2} \right) \right)}{E_0^2 - E_c^2} \right)^{0.5} \qquad (8)$$

$$\beta \doteq 0.4 \alpha Z^{0.6}, \quad U_0 = E_0 / E_c, \quad J \doteq 1.15 \times 10^{-3} Z \qquad (9)$$

where Z and A are the atomic number and atomic weight, η the electron back scatter coefficient and E_0 and E_c are the accelerating potential and critical excitation potential respectively. ϕ_0 and β are the variables most affected by the changing Z for layered versus unlayered samples, their variation with δ, δ' has yet to be determined. A simple linear weighting based on a scale length of $1/2\alpha$ is a reasonable starting point and gives for a surface layer, δ,

$$\beta_{layer} = \delta \cdot 2\alpha \cdot \beta_{matrix} + (1 - \delta \cdot 2\alpha) \cdot \beta_{film} \qquad (10)$$

and

$$\phi_{o\,layer} = \delta \cdot 2\alpha \cdot \phi_{o\,matrix} + (1 - \delta \cdot 2\alpha) \cdot \phi_{film} \qquad (11)$$

In other words, a layer of thickness $\delta = 1/2\alpha$ will in this approximation behave as a semi-infinitely thick sample.

CONCLUSION

An analytical solution has been developed for the problem of the X-ray intensity observed in the microprobe from compositionally stratified samples. This solution will definitely hold for the case of relatively small differences in atomic number. For large differences between layer and substrate the appropriate coefficients will be some weighted mean of the individual values for a semi-infinite solid of composition equal to that of the layer material and the substrate material respectively.

APPENDIX

The integral that results from combining equations 1 and 2 can be integrated by changing the variable.

By definition

$$erf(x) = \frac{2}{\sqrt{\pi}} \int_0^x \exp(-t^2)\,dt \quad \text{or} \quad \int_0^x \exp(-t^2)\,dt = \frac{\sqrt{\pi}}{2} erf(x) \qquad \text{A.1}$$

The integral to be evaluated is of the form:

$$\int_{\delta'}^{\delta} \exp(-a^2x^2 - bx)\,dx = \int_0^{\delta} \exp(-a^2x^2 - bx)\,dx - \int_0^{\delta'} \exp(-a^2x^2 - bx)\,dx \qquad \text{A.2}$$

now $$a^2x^2 + bx = a^2\left(x + \frac{b}{2a^2}\right)^2 - \left(\frac{b}{2a}\right)^2 \qquad \text{A.3}$$

so let $t = \left(ax + \frac{b}{2a^2}\right)$; $dt = a\,dx$.

The integrals become:

$$\frac{\exp\left(\frac{b^2}{4a^2}\right)}{a} \int_{\frac{b}{2a}}^{a\left(\delta + \frac{b}{2a^2}\right)} \exp(-t^2)\,dt - \frac{\exp\left(\frac{b^2}{4a^2}\right)}{a} \int_{\frac{b}{2a}}^{a\left(\delta' + \frac{b}{2a^2}\right)} \exp(-t^2)\,dt \qquad \text{A.4}$$

and can be written as:

$$\rightarrow \frac{\exp\left(\frac{b^2}{4a^2}\right)}{a} \left[\int_0^{a\delta + \frac{b}{2a}} \exp(-t^2)\,dt - \int_0^{\frac{b}{2a}} \exp(-t^2)\,dt - \int_0^{a\delta' + \frac{b}{2a}} \exp(-t^2)\,dt + \int_0^{\frac{b}{2a}} \exp(-t^2)\,dt \right] \qquad \text{A.5}$$

or

$$\rightarrow \frac{\exp\left(\frac{b^2}{4a^2}\right)}{a}\left(\operatorname{erf}\left(a\delta+\frac{b}{2a}\right)-\operatorname{erf}\left(a\delta'+\frac{b}{2a}\right)\right)\exp(-\delta' x') \qquad \text{A.6}$$

The final $\exp(-\delta' x')$ represents the absorption in the over-layer.